

for

POWDER PROCESSING

by

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BACKGROUND OF THE INVENTION

10 This invention relates to forming particulate powdered materials for use, particularly in the pharmaceutical, nutritional and dietary supplement industries. The invention is particularly concerned with providing an end product, which is denser, more granular and generates less dust with better flow than previously known products for these purposes. This is particularly valuable in that the end product has a  
15 minimum of, and in most cases no, fillers or diluents to obtain the desired end product, which is therefore substantially pure.

Traditional densification and dry granulation is performed mainly by the pharmaceutical industry using a roll compactor. An example of this is system known as the Chilsonator<sup>(TM)</sup>. The success of roll compaction is limited by the  
20 compactability or cohesiveness of the specific material. Frequently, the material requires preblending with compressible fillers and diluents in order to achieve compaction. Even with the most suitable fillers, roll compaction is frequently inefficient and slow because not all of the material gets compacted on the first pass through the rollers. Uncompacted material must be constantly recycled back through  
25 the machine until compacted. Compacted material forms a thin brittle ribbon which is then ground back into a powder of desired particle size. The resulting powder is normally higher in density or more compressible. The resulting powder however has fillers or diluents, which are generally undesirable.

An alternate way of accomplishing similar results is by using a tablet press to  
30 compress large tablets or slugs ("slugging") then milling them back into a powder.

This format also has the disadvantage that there are diluents and fillers, which are generally undesirable.

There is, accordingly, a need to provide for improved system of powderized products which can provide enhanced flow properties, and better compaction and compressibility. There is a need to provide such a system which can permit for the production of end products, for instance, in the form bulk powders, or powders for tableting or encapsulating in gelatin capsules or the like.

### SUMMARY OF THE INVENTION

By this invention the Applicants minimize the disadvantages of known techniques for processing raw material products. The Applicants provide a system for forming powderized or granulated products of higher density, with less dust, better granularity and better flow characteristics.

According to the invention, there is a system as provided for pelletizing particulate raw matter, and thereafter subjecting the pellets to a milling process to obtain a powdered form of the particulate. During pelletizing steam can be added. The resulting powder is denser, more granular and has better free flow properties than the pre-pelletized particulate matter. The powderized particulate has applications for pharmaceutical, nutritional and dietary supplement end products.

A system is provided for pelletizing particulate raw matter having a first density, and thereafter subjecting the pellets to a milling process to obtain a powdered form of the particulate. During pelletizing steam can be added. The resulting powder is denser, more granular and has better flow than the pre-pelletized particulate matter having the first density. The powderized particulate has applications for pharmaceutical, nutritional and dietary supplement finished products.

There is apparatus for processing products for increasing the density of particulate matter in a powderized form which comprises feed means for directing particulate matter into a pelletizing mill, the particulate matter being in a first powderized form and having a first density. A pelletizing mill generates pellets of the particulate matter. The pellets are then milled into a second powderized form, whereby the second powderized form of the particulate has a greater density than the

first density. This is achieved in a manner where the formed pellet substantially exclude diluents or fillers.

During pellitization, some products required the introduction of saturated steam at a selected temperature, pressure and condensation characteristic. There is at least about 95% substantially pure saturated steam under a pressure of about 40 to about 80 PSI at about a temperature of about 180°F to about 400°F. This hydrates the particulate matter at a temperature of about 80°F to 200°F and thereby add about 1% moisture to the particulate matter. The product with increased moisture content is forced under pressure through a spinning perforated dye of a predetermined dimension thereby to obtain a pellet of a selected size, the forcing through the dye being effected selectively by dual inner rotating roller means.

There is a pre-milling step for processing raw materials to obtain a particulate matter for feeding into the pelletizing mill. The pellets exposed to steam are cooled to a substantially ambient temperature prior to being milled. A cooler at an outlet from the pelletizing mill permits ambient air to pass through a bed containing pellets discharged from the pelletizing mill.

The processed product is relatively coarser, capable of improved flow and relatively more compressible than the particulate matter. The powderized product in the second form has relatively greater granularity than the particulate matter in the first form. In the second powderized form the product can be made into, selectively, bulk powders such as teas or sports drinks or tablets, and capsules, and selectively have at least one other ingredient.

The invention is further described with reference to the accompanying drawings.

#### BRIEF DESCRIPTION OF THE DESCRIPTION DRAWINGS

FIG. 1 is a flow diagram illustrating a prior art method to form granular materials and tablets according to a dry granulation technique.

FIG. 2 is a flow diagram illustrating a method to form granular materials and tablets according to a dry granulation system of the invention.

## DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

The Applicants describe by example a system, apparatus and method for performing the process of the invention. The invention includes the apparatus for performing the process under the process, and also products made by the apparatus.

5       The system provides for pelletizing particulate raw matter, and thereafter subjecting the pellets to a milling process to obtain a powdered form of the particulate. During pelletizing steam is added. The resulting powder is denser, more granular and has better flow than the pre-pelletized particulate matter. The powdered particulate end product has applications for pharmaceutical, nutritional  
10       and herbal end products.

      There is apparatus and a method for processing products for increasing the density of particulars in a powdered form. Feed means directs particulate matter into a pelletizing mill. The particulate matter has a first density. The pelletizing mill forms pellets of the particulate matter. Thereafter the pellets are milled into a  
15       powderized form. The powdered form of the particulate has a second density which is a greater density than the first density.

      The steam fed pellet mill is used to increase the density of powders, create or enlarge granules, control particle size, enhance flow properties, decrease dust and create granulations without using diluents or fillers. The products are particularly, but  
20       not exclusively, applicable to making end products for the pharmaceutical, nutritional or dietary supplement industries.

### ***General Description of Pelletizing Process:***

      A pellet mill originally designed to make animal feed is used to control  
25       particle size, increase a product's density, or add granularity and decrease dust by forming a pellet and milling it back into powder form.

      Particulate product of predetermined particle size is fed into a conditioning chamber where it can be exposed to saturated steam of controlled temperature, pressure and condensation. The steam increases the product's inherent moisture  
30       content and compressibility. This benefit is normally only obtained through the use of

compressible fillers in the traditional dry granulation methods of the pharmaceutical industry.

To form the pellet, the conditioned product is forced at extreme pressure through a spinning perforated die of preselected dimension by two rotating inner rollers. Nearly all of the material compresses into pellets with a minimal amount of product "fines". The lack of "fines" increases the pellet mill's overall output because there is no need to recycle a large percentage of the product back through the machine. Increased product throughput with minimal recycling makes pelletizing much more cost effective than alternate dry granulation means.

Pellets are quickly cooled to room temperature and milled to a specific particle size range based on the intended application of the product. The resulting powder is coarser, denser, better flowing with less dust, and more compressible or compactable. This makes it ideal for the manufacturing of pharmaceutical dosage forms such as tablets and capsules or nutritional powder drink mixes. This can be a powder blend.

***Pre-milling – (Preparing the raw material for pelletizing)***

Using a hammermill or equivalent milling device for whole plants, roots, leaves, dried fruits or pharmaceutical actives are ground to produce a fine homogeneous powder. Traditionally, the pellet mill is used to manufacture products for the grain or animal feed industry. In this prior art application, molasses, starches and fats are added at the conditioning chamber along with steam to help the material bind together. Required particle size is only about 700 microns (25 mesh) because the binders help hold the pellet together.

Binders used to make animal feed are not desirable in the pelletizing of herbal or pharmaceutical products according to the present invention. Without binders, the particle size of the starting raw material becomes extremely critical. Products are ground to finer particle size, ranging from 100 to 1300 microns (14 to 150 mesh), to increase the surface area and create more binding sites within each individual particle. The compacting step requires more shear force because it is dependent on steam or the product's own compressible properties.

### ***Steam and the conditioning chamber***

Inside the conditioning chamber, the powder bed is typically penetrated by 95% to 100% pure saturated steam with a typical pressure of 40 to 80 PSI. Steam temperature may range between 180°F to 400°F while it heats and hydrates the powder to a temperature of 80°F to 200°F adding approximately 1% moisture. Products that are naturally compressible solely by shear force, require no steam to form a pellet.

### ***Feeding the pellet mill***

The rate at which the conditioning chamber feeds the pellet mill is controlled by the amount of steam and the required heat exposure to form good pellets. Feeding rate is also controlled by resistance created inside the die while pelletizing, product flow and force needed to compress. Ultimately, an optimum flow rate setting is determined for each individual product.

### ***The effect of product variability on the pelletizing process***

Certain products with good compressible properties such as ginger root powder can be pelletized with little or no steam depending on the individual product lot.

Products which contain a high percentage of fibrous matter, such as Siberian Ginseng, require substantial exposure to steam and greater force to compact.

Products with low melting points that compact with minimal force require a relieved die that subjects the product to less shear force and friction. These products will also form pellets without the need for steam.

### ***Characteristics of a Typical Pellet Mill***

25HP Motor 1800 RPM

Die Speed 230 RPM

Mixer Speed 500 RPM

12' die, standard, 3.94" wide 149in<sup>2</sup> Working Area

Density of typical pellet 1.3 g/mL

Length of pellets can vary between  $\frac{1}{4}$  " to 2" depending on the blade setting  
CPM (California Pellet Mills), Series 1100 Pellet Mill or Series 3000 Pellet  
Mill with Counter flow Coolers.

## 5 **Cooler**

Heated pellets are discharged from the pellet mill into the cooler. The cooler  
fan draws ambient air upward through the pellet bed. The pellets are cooled gradually  
as they move through the rising air current. To prevent cooling shock, cold air comes  
in contact with cooled hardened pellets while incoming hot pellets come in contact  
10 with air preheated by the warm pellets before it.

## *Specification in Milling Pellets – Controlling Particle Size and Density*

Pellets are milled to meet a wide range of specifications. Typical ranges can  
be anywhere between 100 and 1300 microns or 100% through 14 to 150 mesh.

15 For raw materials that are going to be compresses into tablets, a normal  
requirement would be 100% through a 14 mesh with a bell shape standard distribution  
of particle sizes and perhaps no more than 40% through a 100 mesh. This would be  
difficult to produce without the pelletizing process because most raw material  
powders would be either too fine or of a homogeneous particle size. The granularity  
20 created by the pelletizing process is what makes the product suitable for tablet  
compression.

For raw materials that are going to be placed inside gelatin capsules, a typical  
particle size requirement can be 100% through a 60 or 80 mesh. Powders that are just  
milled and do not undergo the pelletizing process can sometimes be as fine as 100%  
25 through 200 mesh. These products are dusty and difficult to work with because of  
their poor flow characteristics. The pelletizing process would make this product  
coarser and better flowing.

Added density and no fillers allow product formulators design dosage forms  
with more consumer appeal. Tablets and capsules are smaller, more concentrated or  
30 undiluted and easier to swallow.

### ***Advantages***

Particle size shift to bigger screens on the sieve analysis test – more coarse particles result and less dust or fines.

5 Better particle size distribution – not all particles are nearly the same size.

Same moisture – Steam does not add additional moisture to the final product.

Increased density – Density increase in excess of 100% is possible on some products.

10 Increased fill weight on gelatin capsules – More product fits in a small capsule.

Better flow – Less tablet or capsule weight variability through better flow and compactability.

More granularity – Some products are now compressible without compressible fillers.

15 Increased manufacturing efficiency – Pellet mill offers more product throughput than traditional means of the pharmaceutical industry.

Applicants now set out in Table 1 some example products as illustrated. For each of these raw material products, there is set out the normal powder characteristics and also the pelletized powder characteristics, the latter being in terms of the current invention.

**TABLE 1**  
**HD Powder Line**  
**Example Products**

	Density	Loose	% Gain	Tapped	% Gain	Mesh Size
Ginkgo Leaf HD	Normal Powder	0.29		0.43		
	Pelletized Powder	0.43	48%	0.56	30%	60
Korean Ginseng Root HD	Normal Powder	0.44		0.59		
	Pelletized Powder	0.56	27%	0.75	27%	60
Echinacea Purpurea Herb HD	Normal Powder	0.22		0.40		
	Pelletized Powder	0.50	127%	0.70	75%	60

Gota Kola Herb HD	Normal Powder	0.35		0.50		
	Pelletized Powder	0.56	60%	0.80	60%	60
Valerian Officinalis Root HD	Normal Powder	0.47		0.62		
	Pelletized Powder	0.64	36%	0.80	29%	40
Siberian Ginseng Root HD	Normal Powder	0.45		0.61		
	Pelletized Powder	0.68	51%	0.90	48%	60
Feverfew Herb HD	Normal Powder	0.28		0.41		
	Pelletized Powder	0.53	89%	0.70	71%	40
Astragalus Root HD	Normal Powder	0.36		0.57		
	Pelletized Powder	0.57	58%	0.77	35%	40
Red Raspberry Leaves HD	Normal Powder	0.31		0.49		
	Pelletized Powder	0.38	23%	0.56	14%	80
Foti Root HD	Normal Powder	0.49		0.62		
	Pelletized Powder	0.66	35%	0.80	29%	40
Alfalfa Herb HD	Normal Powder	0.19		0.34		
	Pelletized Powder	0.43	126%	0.53	56%	40
Ginger Root HD	Normal Powder	0.41		0.61		
	Pelletized Powder	0.54	32%	0.75	23%	20
Black Cohosh Root HD	Normal Powder	0.55		0.58		
	Pelletized Powder	0.61	11%	0.73	26%	40
Cat's Claw Root HD	Normal Powder	0.40		0.58		
	Pelletized Powder	0.54	35%	0.70	21%	60
Nettle Root HD	Normal Powder	0.35		0.48		
	Pelletized Powder	0.52	49%	0.66	38%	80
St. John's Wort Herb HD	Normal Powder	0.34		0.42		
	Pelletized Powder	0.48	41%	0.52	24%	40
Echinacea Angustifolia Root HD	Normal Powder	0.25		0.33		
	Pelletized Powder	0.49	96%	0.57	73%	40
Red Clover Tops HD	Normal Powder	0.30		0.43		
	Pelletized Powder	0.36	20%	0.52	21%	80

The Applicants further set out other exemplary data showing the effectiveness of the product in terms of the invention.

In Table 2 a prior art normal powder technique is set out with its effective analysis. The comparative table for the same product is set out in terms of the

present invention in Table 4. Compare the sections labeled Sieve Analysis and Tapped Density.

In Table 3 sets out a normal powder analysis for a different product in terms of the prior art. Table 5 sets out an analysis of the same product in terms of the invention. Also, compare the sections labeled Sieve Analysis and Tapped Density.

**TABLE 2**  
**Normal Powder (Prior Art)**

**10 SIEVE ANALYSIS**

Test Description		Result
% THROUGH US	#40 SCREEN	100.0000
% THROUGH US	#60 SCREEN	98.5000
% THROUGH US	#80 SCREEN	58.9000
15 % THROUGH US	#100 SCREEN	48.3000
% THROUGH US	#140 SCREEN	33.6000

**TABLE 3**  
**Normal Powder (Prior Art)**

**20 SIEVE ANALYSIS**

Test Description		Result
% THROUGH US	#40 SCREEN	100.0000
25 % THROUGH US	#60 SCREEN	99.8000
% THROUGH US	#80 SCREEN	85.0000
% THROUGH US	#100 SCREEN	74.5000
% THROUGH US	#140 SCREEN	56.0000

**TABLE 4**  
**Pelletized Powder (Invention)**

**30 SIEVE ANALYSIS**

Test Description		Result
35 % THROUGH US	#40 SCREEN	100.0000
% THROUGH US	#60 SCREEN	99.3000
% THROUGH US	#80 SCREEN	68.3000
% THROUGH US	#100 SCREEN	55.0000
40 % THROUGH US	#140 SCREEN	30.9000

**TABLE 5**  
**Pelletized Powder (Invention)**

**SIEVE ANALYSIS**

	Test Description	Result
	% THROUGH US #40 SCREEN	100.0000
10	% THROUGH US #60 SCREEN	98.1000
	% THROUGH US #80 SCREEN	70.7000
	% THROUGH US #100 SCREEN	58.2000
	% THROUGH US #140 SCREEN	37.3000

15 Many other forms of the invention exist, each differing from the other in matters of detail only. For instance different feed means can be used for directing particulate matter into the pelletizing mill. Different consistencies of the particulate matter in its first powderized form and first density are possible. Different formats of pelletization are possible. Different pellet mill manufacturers and different pellet mill sizes and die specifications are possible.

20 Although ideally there are no diluents or fillers, it is possible under certain preferred formulations to add a degree of such products. The introduction of steam into the pellet mill may be optional, as indeed are different pressures and temperatures possible. Cooling of the pellets to a substantially ambient temperature prior to being milled by the milling means can be optional. Other cooling temperatures are possible.

25 The increased moisture content can be added by techniques other than forced pressure through a spinning perforated dye, and other than by forcing material through the dye being effected selectively by inner rotating roller means. The pellets can vary in size, consistency and shape. The milled pellets output in a powderized form can have sizes different to a size between about 100 to about 800 microns. Also, the size of the output powder can be different to a size where about 100% of the powderized product is passable through a 14 mesh.

30 The end products of the method and/or apparatus may be for use in industries other than for the pharmaceutical, nutritional or herbal end products.

35 The invention is to be determined solely by the following claims.